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(54) DIE MADE OF W BASED SINTERED ALLOY FOR HOT PRESS MOLDING OF HIGH PRECISION OPTICAL GLASS LENS HAVING EXCELLENT GLASS CORROSION RESISTANCE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a die made of a W based sintered alloy for hot press molding of a high precision optical glass lens which has excellent glass corrosion resistance.

SOLUTION: The die for hot press molding of a high precision optical glass lens consists of a W based sintered alloy having a composition containing 0.2 to 0.8 mass% Ni, and the balance W with inevitable impurities, and has a structure in which a thin alloy layer with Ni diffusedly contained is present along the grain boundaries between the W grains, and a free Ni phase is not present on the grain boundaries.

CLAIMS

[Claim(s)]

[Claim 1]nickel: A presentation which contains 0.2 – 0.8 mass % and the remainder becomes from W and an inevitable impurity, And a metallic mold made from W basis sintered alloy of a high precision optical glass lens excellent in glass corrosion-proof nature constituting from a W basis sintered alloy which has an organization where an alloy thin layer of nickel diffusion content exists along a grain boundary between W grains, and an isolation nickel phase does not exist in said grain boundary for hot pressing shaping.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention]Especially this invention shows the corrosion resistance outstanding to the very powerful corrosive glass of fluoridation glass etc., and. It is related with the metallic mold made from W basis sintered alloy suitable for carrying out hot pressing shaping of the high precision optical glass lens which are the parts of the optical function device which is excellent also in thermal conductivity (heat dissipation nature), and has a still lower coefficient of thermal expansion therefore, with which various kinds of electrons and electric appliances, optical apparatuses, etc. are equipped.

[0002]

[Description of the Prior Art]Conventionally, generally various kinds of stainless steel and heat resisting steel, and the metallic mold further formed with a Co base alloy, a Ni group alloy, etc. are used for hot pressing shaping of an optical glass lens.

[0003]

[Problem(s) to be Solved by the Invention]Highly-efficient-izing and a miniaturization of the electron and electric appliance of various kinds in recent years on the other hand, or an optical apparatus are remarkable, In connection with this, the optical glass lens used for these optical function devices Small-scale-izing and ultrathin-izing, Although it is furthermore in the tendency of highly-precise-izing, and the temperature rise of the hot pressing molding temperature of said optical glass lens is carried out corresponding to this, it is the fluoridation glass thing with which practical use is presented widely and the molding temperature is going to amount also to 900 ** from about 600 **, The actual condition is that the above-mentioned conventional metallic mold cannot perform highly precise-ization to satisfaction enough since corrosion advance is very quick under such elevated-temperature press-forming conditions, the cavity surface (die surface) of a metallic mold is comparatively ruined between short ** and not only resulting in a use life but the coefficient of thermal expansion is relatively large.

[0004]

[Means for Solving the Problem]Then, this invention persons show corrosion resistance excellent also in the bottom of elevated-temperature press-forming conditions from the above viewpoints, And that a metallic mold which enables hot pressing shaping of a high precision optical glass lens should be developed, As a result of inquiring, nickel:0.2 – 0.8 mass % is contained for a metallic mold, If constituted from a W basis sintered alloy which has an organization where an alloy thin layer of nickel diffusion content exists along a presentation which the remainder becomes from W and an inevitable impurity, and a grain boundary between W grains, and an isolation nickel phase does not exist in said grain boundary, Stainless steel and heat

resisting steel in which a metallic mold made from W basis sintered alloy of this result constitutes the above-mentioned conventional metallic mold, As opposed to furthermore a Co base alloy, a Ni group alloy, etc. showing melting point:1450-1550 **, thermal conductivity:0.16-0.22cal/cm-secand**, and coefficient-of-thermal-expansion:11 - 14x10 ^{6}/**, The melting point equivalent to W since W basis sintered alloy which constitutes this has W grain texture : About 3400 **, Thermal conductivity : it not only becomes possible, since it has about 0.48cal/cm-sec, **, and coefficient-of-thermal-expansion:abbreviation 4.5x10 ^{6}/** and a high-melting point, high heat conductivity (high heat dissipation nature), and a low thermal expansion coefficient are provided in this way to attain highly precise-ization of an optical glass lens, but, From that an isolation phase does not exist in a grain boundary of W grain ********** in particular, and corrosion resistance outstanding to fluoridation glass etc. being shown conjointly. A research result of having continued at a long period of time and coming to demonstrate performance excellent in hot pressing shaping of a high precision optical glass lens under elevated-temperature press-forming conditions was obtained.

[0005]This invention is made based on the above-mentioned research result, and 0.2 to nickel:0.8 mass %, Contain and an alloy thin layer of nickel diffusion content exists along a presentation which the remainder becomes from W and an inevitable impurity, and a grain boundary between W grains, It has the feature in a metallic mold made from W basis sintered alloy of a high precision optical glass lens excellent in glass corrosion-proof nature for hot pressing shaping constituted from a W basis sintered alloy which has an organization where an isolation nickel phase does not exist in said grain boundary.

[0006]Having determined a Ni content of W basis sintered alloy which constitutes a metallic mold made from W basis sintered alloy of this invention as 0.2 to 0.8 mass %, Formation of nickel diffusion content alloy thin layer by which the content is formed in a grain boundary between W grains by less than 0.2 mass % at the time of sintering is insufficient, Since adhesion sufficient between W grains is not securable, if desired high intensity cannot be secured to a metallic mold but the content exceeds 0.8 mass % on the other hand, nickel isolation phase comes to appear in a grain boundary of W grain, and this is based on a reason for coming to bring about a remarkable fall of glass corrosion-proof nature.

[0007]

[A mode of implementation of an invention] Below, an example explains concretely a metallic mold made from W basis sintered alloy of this invention. As the end of precursor powder, prepare W powder with mean particle diameter of 3.2 micrometers, and the 0.7micrometer nickel nitrate powder, blend the end of these precursor powder with a predetermined rate, and wet blending is carried out in a ball mill for 48 hours using an acetone solvent, It is considered as W powder by which surface coating was carried out with said nickel nitrate which dissolved in said acetone solvent, Subsequently, heat-treat [800 **] to W powder of said nickel nitrate covering on condition of maintenance among a hydrogen atmosphere for 1 hour, and the pyrolysis of the nickel nitrate on W powder surface is carried out to it, The surface considers it as W powder covered with ultra-thin nickel layer, fills up a rubber mold with this, and carries out press forming with hydrostatic pressure of 150MPa, diameter: -- 50 mmx height: -- a Plastic solid with a size of 40 mm being formed, and to this Plastic solid. A glost firing join and also temperature in conditions of 1450 ** 1-hour maintenance among presintering in conditions of 900 ** 5-hour maintenance among a hydrogen atmosphere, and a hydrogen atmosphere : Hot isostatic press processing on conditions of 1300 ** and pressure:100MPa

is performed, diameter: --- 40 mmx length: --- considering it as a size of 30 mm, and a die material with a presentation shown in Table 1, respectively --- these die materials --- two pieces being used as one pair of fluctuated types, respectively, and a diameter:38mmx central part depth:5mm curved-surface cavity on the upper surface of a bottom part of these, [form and] On the other hand, by considering the undersurface of a punch as as [flat surface], and grinding a these both upper-and-lower-sides type cavity surface to surface roughness not more than Rmax:0.05micrometer, a metallic mold made from this invention W basis sintered alloy. (It is hereafter called this invention metallic mold) 1-7, and a Ni content manufactured each for a metallic mold made from a comparison W basis sintered alloy (henceforth a comparison metallic mold) which separated in the higher one from the scope of this invention. When an organization of this invention metallic molds 1-7 obtained as a result and a comparison metallic mold is observed using an optical microscope (400 times), this invention metallic molds 1-7, Although all made W grain a subject and an organization where an isolation nickel phase does not exist was shown only by an alloy thin layer of nickel diffusion content existing in a mutual grain boundary of said W grain, an organization where an isolation nickel phase small to a grain boundary between W grains carried out distributed distribution was looked at by comparison metallic mold.

[0008]A Co base alloy molten metal and 13 thirteen-Cr-stainless-steel molten metal which had the component composition shown in Table 1, respectively for the comparative purpose are prepared, this molten metal --- diameter: --- 120 mmx length: --- it casting to a 200-mm ingot, and hot forging being started and this with cooking temperature at 1100 **, diameter: --- 40 mmx length:, after considering it as a forge raw material with a size of 30 mm and homogenizing on condition of after-maintenance air cooling for 15 minutes for this forge raw material subsequently to 1000 **, Use two each of these forge raw material as one pair of fluctuated types, and a diameter:38mmx central part depth:5mm curved-surface cavity is formed in the upper surface of a bottom part of these, On the other hand, the undersurface of a punch was considered as as [flat surface], and manufactured each for the optical glass lens press-forming public-funds types (conventionally henceforth a metallic mold) 1 and 2 conventionally by grinding a these both upper-and-lower-sides type cavity surface to surface roughness not more than Rmax:0.05micrometer.

[0009]Next, about these metallic molds of various kinds of, as a cob which is a glass lens raw material by capacity %. BaF_2: 41%, aluminum$(PO_3)_3$:14%, SrF_2: 12%, AlF_3:10%, $Ba_2P_2O_7$:8%, Contain and the remainder uses fluoridation glass with presentation, aluminum$_2O_3$, ** and others, Capacity per one of said gob : 0.2-cm^3, cooking temperature:900 ** of said gob, Press-forming pressure: Press forming of an optical glass lens was performed on condition of for 10MPa and press-forming speed:six-piece/, and the lens shaping number until surface roughness of a cavity surface amounts to Rmax:0.06micrometer was measured. Similarly this measurement result was shown in Table 1.

[0010]

[Table 1]

種別		成分組成(質量%)					レンズ成形個数(個)
		Ni	Cr	Fe	Co+不純物	W+不純物	
本発明金型	1	0.21	—	—	—	残	15600
	2	0.32	—	—	—	残	16100
	3	0.40	—	—	—	残	15300
	4	0.53	—	—	—	残	15200
	5	0.61	—	—	—	残	15100
	6	0.69	—	—	—	残	15200
	7	0.78	—	—	—	残	14000
比較金型		0.93※	—	—	—	残	510
従来金型	1	—	13.3	残(+不純物)	—	—	113
	2	—	9.4	28.5	残	—	94

(表中、※印は本発明範囲外を示す)

[0011]

[Effect of the Invention]From the result shown in Table 1, this invention metallic molds 1-7, All excel [subject / of W basis sintered alloy which constitutes this] in glass corrosion-proof nature, And it consists of a high-melting point, high heat conductivity (high heat dissipation nature), and a W grain that has a low thermal expansion coefficient, And with having an organization where only the alloy thin layer of nickel diffusion content which has the characteristic which said W grain has, and the equivalent outstanding characteristic exists in the mutual grain boundary of said W grain conjointly, The isolation nickel phase which carries out distribution-on organization distribution in a comparison metallic mold to the corrosion advance by the heating-at-high-temperature fluoridation glass gob of a cavity surface being controlled remarkably, and continuing at a long period of time and holding a good cavity surface by a cause. Conventionally in [the corrosion of a cavity surface advances and] the metallic molds 1 and 2, It is in ** that the surface roughness of a cavity surface falls comparatively for a short time, and it becomes impossible to present practical use from especially the whole organization of a cavity surface being corroded with the very strong corrosive fluoridation glass lens heated by the elevated temperature. As mentioned above, the metallic mold made from W basis sintered alloy of this invention, For example, not to mention hot pressing shaping of the optical glass lens using corrosive weak silica glass, boronizing glass, etc., comparatively, Since the corrosion resistance outstanding also in heating-at-high-temperature press forming, such as powerful, especially corrosive fluoridation glass, is shown, they are small-scale-izing of an optical glass lens and ultrathin-izing, and a thing that can respond to highly precise-ization enough further at satisfaction.

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Ｆターム(参考)　４Ｋ０１８ ＡＡ２０ ＢＡ０９ ＢＣ３２ ＣＡ２３ ＥＡ１２
ＫＡ１８ ＫＡ５８

(54)【発明の名称】　耐ガラス腐食性にすぐれた高精度光学ガラスレンズの熱間プレス成形用Ｗ基焼結合金製金型

(57)【要約】
【課題】　耐ガラス腐食性にすぐれた高精度光学ガラスレンズの熱間プレス成形用Ｗ基焼結合金製金型を提供する。
【解決手段】　高精度光学ガラスレンズの熱間プレス成形用金型を、Ｎｉ：０．２～０．８質量％、を含有し、残りがＷと不可避不純物からなる組成、並びにＷ粒相互間の粒界にそってＮｉ拡散含有の合金薄層が存在し、前記粒界に遊離Ｎｉ相が存在しない組織を有するＷ基焼結合金で構成する。

【特許請求の範囲】

【請求項１】　Ｎｉ：０．２～０．８質量％、を含有し、残りがＷと不可避不純物からなる組成、並びにＷ粒相互間の粒界にそってＮｉ拡散含有の合金薄層が存在し、前記粒界に遊離Ｎｉ相が存在しない組織を有するＷ基焼結合金で構成したことを特徴とする耐ガラス腐食性にすぐれた高精度光学ガラスレンズの熱間プレス成形用Ｗ基焼結合金製金型。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】この発明は、特に弗化ガラスなどの腐食性のきわめて強いガラスに対してすぐれた耐腐食性を示すと共に、熱伝導性（放熱性）にもすぐれ、さらに低い熱膨張係数を有し、したがって例えば各種の電子・電気機器や光学装置などに装着されている光機能装置の部品である高精度光学ガラスレンズを熱間プレス成形するのに適したＷ基焼結合金製金型に関するものである。

【０００２】

【従来の技術】従来、一般に光学ガラスレンズの熱間プレス成形には、各種のステンレス鋼や耐熱鋼、さらにＣｏ基合金やＮｉ基合金などで形成された金型が用いられている。

【０００３】

【発明が解決しようとする課題】一方、近年の各種の電子・電気機器や光学装置の高性能化および小型化はめざましく、これに伴い、これらの光機能装置に用いられている光学ガラスレンズも小寸化および極薄肉化、さらに高精度化の傾向にあり、これに対応して前記光学ガラスレンズの熱間プレス成形温度は高温化し、広く実用に供されている弗化ガラス製のもので、その成形温度は約６００℃から９００℃にも達しようとしているが、上記の従来金型は、このような高温プレス成形条件下では腐食進行がきわめて速く、比較的短時間で金型のキャビティ面（成形面）が荒れ、使用寿命に至るばかりでなく、熱膨張係数が相対的に大きいために、高精度化を十分満足に行なうことができないのが現状である。

【０００４】

【課題を解決するための手段】そこで、本発明者らは、上述のような観点から、高温プレス成形条件下でもすぐれた耐腐食性を示し、かつ高精度光学ガラスレンズの熱間プレス成形を可能とする金型を開発すべく、研究を行った結果、金型を、Ｎｉ：０．２～０．８質量％、を含有し、残りがＷと不可避不純物からなる組成、並びにＷ粒相互間の粒界にそってＮｉ拡散含有の合金薄層が存在し、前記粒界に遊離Ｎｉ相が存在しない組織、を有するＷ基焼結合金で構成すると、この結果のＷ基焼結合金製金型は、上記の従来金型を構成するステンレス鋼や耐熱鋼、さらにＣｏ基合金やＮｉ基合金などが、融点：１４５０～１５５０℃、熱伝導度：０．１６～０．２２ｃａｌ／ｃｍ・ｓｅｃ・℃、および熱膨張係数：１１～１４×１０$^{-6}$／℃を示すのに対して、これを構成するＷ基焼結合金が、Ｗ粒集合組織をもつことから、Ｗと同等の融点：約３４００℃、熱伝導度：約０．４８ｃａｌ／ｃｍ・ｓｅｃ・℃、および熱膨張係数：約４．５×１０$^{-6}$／℃を有し、このように高融点、高熱伝導性（高放熱性）、および低熱膨張係数を具備することから、光学ガラスレンズの高精度化を図ることが可能となるばかりでなく、特にＷ粒集合組織にの粒界に遊離相が存在しないことと相俟って、弗化ガラスなどに対してすぐれた耐腐食性を示すことから、高温プレス成形条件下での高精度光学ガラスレンズの熱間プレス成形にすぐれた性能を長期に亘って発揮するようになる、という研究結果を得たのである。

【０００５】この発明は、上記の研究結果に基づいてなされたものであって、Ｎｉ：０．２～０．８質量％、を含有し、残りがＷと不可避不純物からなる組成、並びにＷ粒相互間の粒界にそってＮｉ拡散含有の合金薄層が存在し、前記粒界に遊離Ｎｉ相が存在しない組織を有するＷ基焼結合金で構成してなる、耐ガラス腐食性にすぐれた高精度光学ガラスレンズの熱間プレス成形用Ｗ基焼結合金製金型に特徴を有するものである。

【０００６】なお、この発明のＷ基焼結合金製金型を構成するＷ基焼結合金のＮｉ含有量を０．２～０．８質量％と定めたのは、その含有量が０．２質量％未満では、焼結時にＷ粒相互間の粒界に形成されるＮｉ拡散含有合金薄層の形成が不十分で、Ｗ粒相互間に十分な密着性を確保することができないことから、所望の高強度を金型に確保することができず、一方その含有量が０．８質量％を越えると、Ｗ粒の粒界にＮｉ遊離相が出現するようになり、これが耐ガラス腐食性の著しい低下をもたらすようになる、という理由によるものである。

【０００７】

【発明の実施の態様】つぎに、この発明のＷ基焼結合金製金型を実施例により具体的に説明する。原料粉末として、３．２μｍの平均粒径をもったＷ粉末、および同０．７μｍの硝酸ニッケル粉末を用意し、これら原料粉末を所定の割合に配合し、アセトン溶媒を用いてボールミル中にて４８時間湿式混合して、前記アセトン溶媒に溶解した前記硝酸ニッケルで表面被覆されたＷ粉末とし、ついで前記硝酸ニッケル被覆のＷ粉末に、水素雰囲気中、８００℃に１時間保持の条件で加熱処理を施し、Ｗ粉末表面上の硝酸ニッケルを熱分解して、表面が極薄のＮｉ層で被覆されたＷ粉末とし、これをゴム鋳型に充填し、１５０ＭＰａの静水圧にてプレス成形して、直径：５０ｍｍ×高さ：４０ｍｍの寸法をもった成形体を形成し、この成形体に、水素雰囲気中、９００℃に５時間保持の条件での予備焼結、および水素雰囲気中、１４５０℃に１時間保持の条件での本焼結、さらに温度：１３００℃、圧力：１００ＭＰａの条件での熱間静水圧プ

レス処理を施して、直径：４０mm×長さ：３０mmの寸法、およびそれぞれ表１に示される組成をもった金型素材とし、これら金型素材のそれぞれ２個を１対の上下型とし、このうちの下型の上面に直径：３８mm×中心部深さ：５mmの曲面キャビティを形成し、一方上型の下面は平面のままとし、これら両上下型のキャビティ面をＲｍａｘ：０．０５μm以下の面粗度に研磨することにより本発明W基焼結合金製金型（以下、本発明金型という）１～７、およびＮｉ含有量がこの発明の範囲から高い方に外れた比較W基焼結合金製金型（以下、比較金型という）をそれぞれを製造した。なお、この結果得られた本発明金型１～７および比較金型の組織を、光学顕微鏡（４００倍）を用いて観察したところ、本発明金型１～７は、いずれもW粒を主体とし、前記W粒の相互間粒界にＮｉ拡散含有の合金薄層が存在するだけで、遊離Ｎｉ相が存在しない組織を示すものであったが、比較金型にはW粒相互間の粒界に小さな遊離Ｎｉ相が分散分布した組織が見られた。

【０００８】また、比較の目的で、それぞれ表１に示される成分組成をもったＣｏ基合金溶湯および１３Ｃｒステンレス鋼溶湯を調製し、この溶湯を直径：１２０mm×長さ：２００mmのインゴットに鋳造し、これを１１００℃の加熱温度で熱間鍛造を開始して、直径：４０mm×長さ：３０mmの寸法をもった鍛造素材とし、ついでこの鍛造素材に１０００℃に１５分間保持後空冷の条件で均質化処理を施した後、これら鍛造素材のそれぞれ２個を１対の上下型とし、このうちの下型の上面に直径：３８mm×中心部深さ：５mmの曲面キャビティを形成し、一方上型の下面は平面のままとし、これら両上下型のキャビティ面をＲｍａｘ：０．０５μm以下の面粗度に研磨することにより従来光学ガラスレンズプレス成形用金型（以下、従来金型という）１，２をそれぞれを製造した。

【０００９】つぎに、これらの各種の金型について、ガラスレンズ素材であるコブとして、容量％で、BaF_2：４１％、$Al(PO_3)_3$：１４％、SrF_2：１２％、AlF_3：１０％、$Ba_2P_2O_7$：８％、を含有し、残りがAl_2O_3、からなる組成をもった弗化ガラスを用い、前記コブの１個当たりの容量：０．２cm^3、前記コブの加熱温度：９００℃、プレス成形圧力：１０MPa、プレス成形速度：６個／分の条件で光学ガラスレンズのプレス成形を行ない、キャビティ面の面粗度がＲｍａｘ：０．０６μmに達するまでのレンズ成形個数を測定した。この測定結果を同じく表１に示した。

【００１０】

【表１】

種別		成分組成(質量%)					レンズ成形個数(個)
		Ni	Cr	Fe	Co+不純物	W+不純物	
本発明金型	1	0.21	—	—	—	残	15800
	2	0.32	—	—	—	残	16100
	3	0.40	—	—	—	残	15300
	4	0.53	—	—	—	残	16200
	5	0.61	—	—	—	残	15100
	6	0.69	—	—	—	残	15200
	7	0.78	—	—	—	残	14000
比較金型		0.93※	—	—	—	残	510
従来金型	1	—	13.3	残(+不純物)	—	—	113
	2	—	9.4	28.5	残	—	94

(表中、※印は本発明範囲外を示す)

【００１１】

【発明の効果】表１に示される結果から、本発明金型１～７は、いずれもこれを構成するW基焼結合金の主体が耐ガラス腐食性にすぐれ、かつ高融点、高熱伝導性（高放熱性）、および低熱膨張係数を有するW粒からなり、かつ前記W粒の相互粒界には前記W粒のもつ特性と同等のすぐれた特性を有するＮｉ拡散含有の合金薄層しか存在しない組織をもつことと相俟って、キャビティ面の高温加熱弗化ガラスゴブによる腐食進行が著しく抑制され、良好なキャビティ面を長期に亘って保持するのに対して、比較金型では組織上分散分布する遊離Ｎｉ相が原因で、キャビティ面の腐食が進行し、また従来金型１，２においては、キャビティ面の組織全体が特に高温に加熱された腐食性のきわめて強い弗化ガラスレンズによって腐食されることから、比較的短時間でキャビティ面の面粗度が低下し、実用に供することができなくなることが明かである。上述のように、この発明のW基焼結合金製金型は、例えば比較的腐食性の弱い珪酸ガラスや硼化

ガラスなどを用いた光学ガラスレンズの熱間プレス成形は勿論のこと、特に腐食性の強い弗化ガラスなどの高温加熱プレス成形においてもすぐれた耐腐食性を示すものであるから、光学ガラスレンズの小寸化および極薄肉化、さらに高精度化に十分満足に対応できるものである。